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(54) Title of the invention Method of manufacturing lightweight calcium silicate product

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**Detailed description****1. Title of the invention**

Method of manufacturing lightweight calcium silicate product

**2. Claims**

The method of manufacturing lightweight calcium silicate products to provide a lightweight calcium silicate articles produced by molding a specific shape out of raw material containing 0 - 50 weight % of slag, 0 - 50 weight % of gypsum, 5 - 80 weight % of cement and/or slaked lime, 2 - 30 weight % of fiber, 0 - 20 weight % of solid particles composed of alkali-soluble silicon oxide and 5 - 90 weight % of hollow particles composed of the alkali-soluble silicon oxide.

**3. Detailed description of the invention****[Applicable field in industry]**

This invention relates to the method of manufacturing a lightweight cement product, in particular, of a lightweight product with sophisticated design to improve the flexural strength and shrinkage using hollow silicon oxide material.

**[Conventional technology]**

Conventionally the use of pearlite and styrene beads were suggested for ceramic building materials

for the purpose of weight reduction in Pat. Pub. S 48-25718 and Pat. Pub. S47-35061. However, those materials are not strong enough because they do not have the alkali reactivity and they function only as the lightweight aggregate.

[Problem that the invention will solve]

The purpose of this invention is to solve the abovementioned disadvantages of the conventional technology and to provide the method of manufacturing lightweight calcium silicate in order to produce articles with excellent workability in designing and improved flexural strength and shrink properties.

[Means to solve the problem]

This invention aims to solve the abovementioned problems and provides the method of manufacturing lightweight calcium silicate products to provide lightweight calcium silicate articles produced by specifically molding a specific shape out of the raw material containing 0 - 50 weight % of slag, 0 - 50 weight % of gypsum, 5 - 80 weight % of cement and/or slaked lime, 2 - 30 weight % of fiber, 0 - 20 weight % of solid particles composed of alkali-soluble silicon oxide and 5 - 90 weight % of hollow particles composed of the alkali-soluble silicon oxide.

In this invention, the slag contained in the raw material has the following advantage although it is not an essential ingredient: The reaction of  $Al_2O_3$  in the slag facilitates the formation of tobermorite and improves the flexural strength and resistance to freezing and thawing. However, the content should not preferably exceed 50 weight % because  $H_2S$  and other toxic gases arise excessively in the autoclave. More preferable value of slag content should be 30 - 50 weight %.

Gypsum has the following advantage although it is not an essential ingredient. It improves the plasticity of the material and reduces chipping and other defects. However, if the content of gypsum exceeds 50 weight %, the following undesirable conditions occur: Intermediate products such as ettringite and mono-sulfate are formed and it becomes difficult for tobermorite to be formed even in the end product. More preferable value of gypsum content should be 2 - 10 weight %.

In this invention, cement and slaked lime function as follows:  $Ca(OH)_2$  formed in the hydration reaction of cement or slaked lime  $Ca(OH)_2$  causes hydrothermal reaction with  $SiO_2$  and generates C-S-H and tobermorite. These C-S-H and tobermorite are excellent in durability and flexural strength. If the content of the cement and/or slaked lime is less than 5 weight %, the abovementioned effect of the additives is small, and if it exceeds 80 weight %, then it is not desirable because of the following reason: If the content is 5 weight % or less, most of  $SiO_2$  remains unreacted and the flexural strength becomes low. On the other hand, if it exceeds 80 weight %, most of  $Ca(OH)_2$  remains unreacted and the material becomes more vulnerable to carbonation caused by  $CO_2$ , which reduces the durability. The contents of the cement and/or slaked lime should preferably be between 40 and 60

weight %, within the abovementioned potential range. Applicable types of cement would include Portland cement, alumina cement, sulfate-resisting cement, blast furnace cement, pozzolan cement, out of which, Portland cement and alumina cement are desirable because they contain less  $\text{CaSO}_4$ , and they are not likely to generate intermediate products of ettringite or mono-sulfate and are ready to form tobermorite easily.

Fibers are effective in increasing the strength of the product. The content of fibers should not preferably be less than 2 weight % because that effect becomes low. It should not preferably exceed 30 weight % because the surface property would deteriorates and, if it happens to become worse, cracks may arise. More preferable value of fiber content should be 5 - 10 weight %, within the abovementioned range.

Solid particles composed of alkali-soluble silicon oxide have the following advantage when contained although it is not an essential ingredient: When typical  $\text{SiO}_2$  particles are used, the reaction ratio is limited to 30 - 50%. On the other hand, when hollow particles are used, the reaction ratio increases and the amount of unreacted  $\text{SiO}_2$  reduces. This also enables weight reduction. However, if the solid particle content exceeds 20 weight %, an undesirable condition occurs as: The solubility of the solid particles in the boiling water at  $100^\circ\text{C}$  should preferably be 100 ppm or higher. Specific examples of these would include silica fume, diatom earth, white clay and silica sand.

In this invention, hollow particles composed of alkali-soluble silicon oxide not only form air bubbles in concrete but they also elute and penetrate into the concrete to increase the concrete strength. The elution of some hollow particles into concrete during the hardening process would be sufficient, but the elution of all particles would be more preferable. The desirable size of the hollow particles should be within the range of 50 - 500  $\mu\text{m}$ . Particles smaller than this range are not desirable because the bulk specific gravity becomes higher, which makes it difficult to reduce the weight. Particles larger than this range are not desirable because a significant floatation phenomenon appears at the time of dehydrating press, and those particles deform or collapse at the time of pressure forming. Desirable thickness of hollow particles should be 1 - 5  $\mu\text{m}$ . If the content of the hollow particles is less than 5 weight %, hardening caused by the additives is not much and weight reduction is not sufficient and flexural strength is low therefore not desirable, while if it exceeds 90 weight %, the flexural strength becomes lower therefore not desirable. More preferable content is within 20 - 80 weight %, in particular, within the range of 30 - 50 weight %. Examples of applicable hollow particles would include shirasu (or sirasu) balloons, silica balloons and glass balloons.

For molding process, materials are blended in the specified proportion and slurried with added water. Then, this slurry is molded into a specific shape using dehydrating press molding, extrusion molding or other process. To evenly disperse hollow particles, extrusion molding should preferably be adopted. To float hollow particles on the surface, however, to ensure specifically sophisticated

design, dehydrating press molding should preferably adopted.

[Embodiment]

Slurry with the composition shown in Table 1 was prepared by blending materials. Then, 1% of methylcellulose was added and a calcium silicate plate was prepared by extrusion molding. The flexural strength, Young's modulus, bulk specific gravity and dimensional change ratio of the calcium silicate plate were measured. The results are recorded in the same table. A comparative example is also included in the table.

The table reveals that the results of this invention have greater flexural strength, smaller dimensional change and smaller bulk specific gravity. The dimensional change is measured according to JIS A-5422.

[Effects of the Invention]

The current invention provides effective improvement in flexural strength, expansion and shrinkage and resistance against freezing and thawing. What is more, the invention has the effect of weight reduction by using solid alkali-soluble silicon oxide. For dehydrating molding, the unique texture of particles can be exposed visible on the surface of the product by floating the hollow particles of low bulk specific gravity.

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Table 1

	Embodiment	1	2	3	4	5	6	7	8	9	10	Comparative example
Composition (weight %)	Blast furnace slag	10	10	10		10	10	10	10			10
	Cement	30	30	30	30	30	40	40	40			40
	Slaked lime									40	40	23
	Silica sand	10	10	10	10	10						10
	Silica fume	4	4	4	14	4	4	4	4	14	14	14
	Shirasu balloon	40	40	40	40		40	40	40	40		0
	Silica balloon					40					40	0
	Bulb (NBKP)	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5	5.5
	Alkali-resistant glass fiber	0.5			0.5	0.5	0.5			0.5	0.5	0.5
	Polypropylene fiber		0.5					0.5				1
	Acrylic fiber			0.5					0.5			
Properties	Flexural strength (kg/cm <sup>2</sup> )	100	97	103	115	92	110	111	107	105	100	88
	Young's modulus (10 <sup>4</sup> kg f/cm <sup>2</sup> )	4.99	4.80	5.01	4.86	4.68	5.09	5.11	5.14	5.05	5.01	4.41
	Bulk specific gravity	0.854	0.850	0.851	0.860	0.851	0.861	0.859	0.863	0.851	0.849	0.852
	Dimensional change (%)	0.113	0.122	0.114	0.092	0.139	0.133	0.137	0.132	0.178	0.182	0.2